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Short communication

Micromixing efficiency of a novel rotor–stator reactor

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Abstract

The importance of inlet region of rotating packed bed (RPB) was confirmed recently. This enlightened us to design another novel reactor, named rotor–stator reactor (RSR), which has multiple inlet regions along radial direction. The micromixing efficiency of RSR was studied by adopting the iodide–iodate reaction as working system. The effects of operating conditions (e.g. rotor speed and reagent concentration) on micromixing efficiency (characterized by segregation index X_S) were investigated. In addition, the effects of different rotor–stator combinations as well as the amount of rotor- and stator-ring on micromixing efficiency were also considered. Based on the incorporation model, the micromixing time of RSR was estimated to reach about 10^{-5} s, which is less than that of rotating packed bed (about 10^{-4} s).

Keywords: Rotor–stator reactor (RSR); Micromixing; Segregation index; Iodide–iodate reaction system; Incorporation model

1. Introduction

Micromixing of reactors have a large influence on the product distribution of a fast chemical reaction. For example, micromixing may influence crystal size distribution and average size of crystal. In polymerization, the molecular weight distribution was also depended on micromixing efficiency of the reactor [1]. Therefore, the development of new reactor provided with excellent micromixing efficiency is necessary and popular.

Micromixing efficiency of many reactors have been studied, including centrifugal pump [2], Tee mixers [3], tubular reactor [4], rotor–stator mixers [5,6], aerated stirred tank [7], couette flow reactor [8], static mixer [9] and so on. Twenty years ago, Ramshaw [10] highlighted rotating packed bed (RPB), a novel reactor that utilizes centrifugal acceleration to intensify mixing and mass transfer. So far, RPB has been applied to desorption [11], absorption [12], distillation [13], ozone oxidation [14] and reactive crystallization [15–17], etc. Our recent research on micromixing efficiency of RPB confirmed that inlet region of RPB plays a very important role in the mixing and reaction [18]. This result enlightened us to design another novel reactor, named rotor–stator reactor (RSR), which has multiple inlet regions arranged in series along radial direction and was expected to exhibit better micromixing efficiency.

In present work, the basic structure and characteristics of RSR was introduced first, and then the effect of operational conditions and different structures on micromixing efficiency was investigated by employing iodide–iodate reaction system. In addition, based on the incorporation model, the micromixing time of RSR was estimated according to our experimental data.

2. Experiments

2.1. Basic structure and characteristics of RSR

The basic structure of RSR was schematically described in Fig. 1. In a RSR, the rotor consists of five rings (named rotor-ring) fixed concentrically on the rotor-seat, which is driven by a motor at a speed ranging from 150 to 2400 rpm (controlled by a frequency inverter); while the stator consists of four rings (named stator-ring) mounted concentrically on the cover cap. The opened slots or holes around the circular of rotor-ring and stator-ring formed channels of fluids. Three different rotors with straight, curve slots or holes around the circular of rotor-rings were used in experiments, they were named RS,

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Nomenclature

A	solution containing $H_2BO_3^-$, I^-, IO_3^-
B	H_2SO_4 solution
C_j	concentration of species j (mol L^{-1})
N	rotor speed (rpm)
n_j	molar amount of species j
X_S	segregation index
Y	selectivity of acid
Y_{ST}	selectivity at the total segregation

Greek letters

τ_m	micromixing time (s)
τ_r	characteristic reaction time (s)

RC and RH, respectively (Fig. 2(a)). Two different stators with straight and curve slots around the circular of stator-rings were used in experiments, they were named SS and SC, respectively (Fig. 2(b)). Therefore, six rotor–stator combinations (named RS–SS, RS–SC, RC–SS, RC–SC, RH–SS, RH–SC) were used in experiments. The inner diameter of each rotor-ring and corresponding stator-ring was different, which was 52, 80, 108, 136, 164 mm for rotor-ring and 66, 94, 122 and 150 mm for stator-rings, respectively. In radial direction, the rotor-rings and stator-rings were arranged in an interval sequence. The thickness in radial direction of each ring was 6 mm, so the gap in radial direction between each rotor-ring and corresponding stator-ring was 1 mm. Aside from being specially announced, the amount of rotor- and stator-rings is respectively 5 and 4 in context, and the rotor–stator combination used is RS–SS because of its simple structure and easy to be manufactured.

As mentioned above, the main characteristics of RSR were as follows: (a) the rotor- and stator-rings were arranged in an interval sequence in radial direction, which consists of multiple inlet regions along radius direction. The fluid was redistributed when passing through every stator-ring; (b) fluid was fully dispersed when passing through the slots or holes opened on rotor-rings and stator-rings, so high surface renewing ratio of fluid elements can be obtained; (c) the channel of fluid can not be fouled because fluid stands high shear force when passing through the gaps between the rotor-ring and corresponding stator-ring. It is suitable for high viscous or crystal system; (d) RSR has excellent flexibility because the rings were fixed on cover cap and rotor-seat by bolts, which is easy to dismantle and assemble or replace according to specific demands.

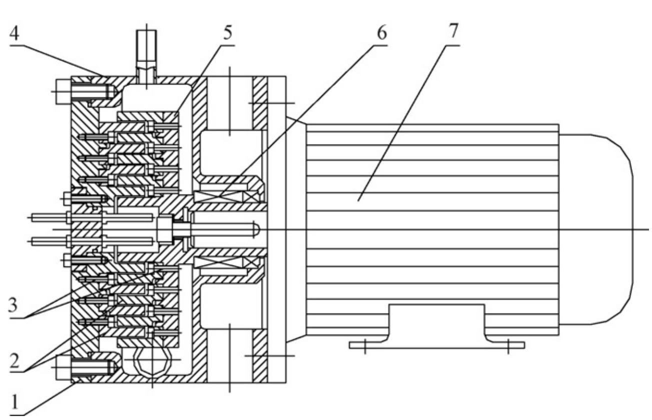

Fig. 1. Schematic description of RSR. (1) Cover cap; (2) rings; (3) bolts; (4) casing; (5) rotor-seat; (6) seal; (7) motor.

2.2. Parallel competing reaction system

Physical methods, such as optical and conductometric methods have been limited because of their poor distinguishability and instrumental limitations. Hence, chemical methods (i.e. chemical reactions as molecular probes) were widely used to test the micromixing efficiency. Fournier et al. developed a novel parallel competing reaction scheme used as working system to characterize micromixing [19]. Its experimental procedure and reactions kinetics were also presented [20,21]. This reaction scheme consists of the following three chemical reactions:

$$H_2BO_3^- + H^+ \Leftrightarrow H_3BO_3 \tag{1}$$

$$5I^- + IO_3^- + 6H^+ \Leftrightarrow 3I_2 + 3H_2O \tag{2}$$

$$I^- + I_2 \Leftrightarrow I_3^- \tag{3}$$

The neutralization reaction (1) is quasi-instantaneous, and redox reaction (2) is fast but much slower than the reaction (1). In perfect mixing conditions, the injected H^+ was consumed by reaction (1), and reactions (2) and (3) may not occur. In partial or total segregation conditions, the injected H^+ was consumed competitively by reactions (1) and (2), and the formed I_2 can further react with I^- to yield I_3^- according to reaction (3). The amount of I_3^- produced depends on the micromixing efficiency. The selectivity Y of H^+ and the segregation index X_S were defined respectively:

$$Y = \frac{2(n_{I_2} + n_{I_3^-})}{n_{H_0^+}} \tag{4}$$

$$X_S = \frac{Y}{Y_{ST}} \tag{5}$$

where $Y_{ST} = 6C_{IO_{3,0}^-}/(6C_{IO_{3,0}^-} + C_{H_2BO_{3,0}^-})$ is the value of Y in total segregation case. The value of X_S is within the range of $0 < X_S < 1$ for partial segregation, and $X_S = 0$ and $X_S = 1$ indicate perfect micromixing and total segregation, respectively.

In our experiments, solutions A and B were prepared first. The procedure for preparation of solution A was as follows: (1) powders H_3BO_3 (281.017 g) and NaOH (90.909 g) were dissolved in 15 and 5 L water, respectively. And then the as-prepared solutions were mixed to obtain the buffer solution; (2) powders KI (48.42 g) and KIO_3 (12.483 g) were dissolved in 0.5 and 2.5 L water, respectively. And then these two solutions were added to the said buffer solution; (3) the rest water was added into the as-prepared solution to prepare 25 L of solution A, which was a mixture of iodate (0.01167 mol L^{-1}), iodide (0.00233 mol L^{-1}) and borate ions (0.0909 mol L^{-1}). The solution B was H_2SO_4 solution (0.05–0.08 mol L^{-1}, corresponding concentration of H^+ was 0.10–0.16 mol L^{-1}).

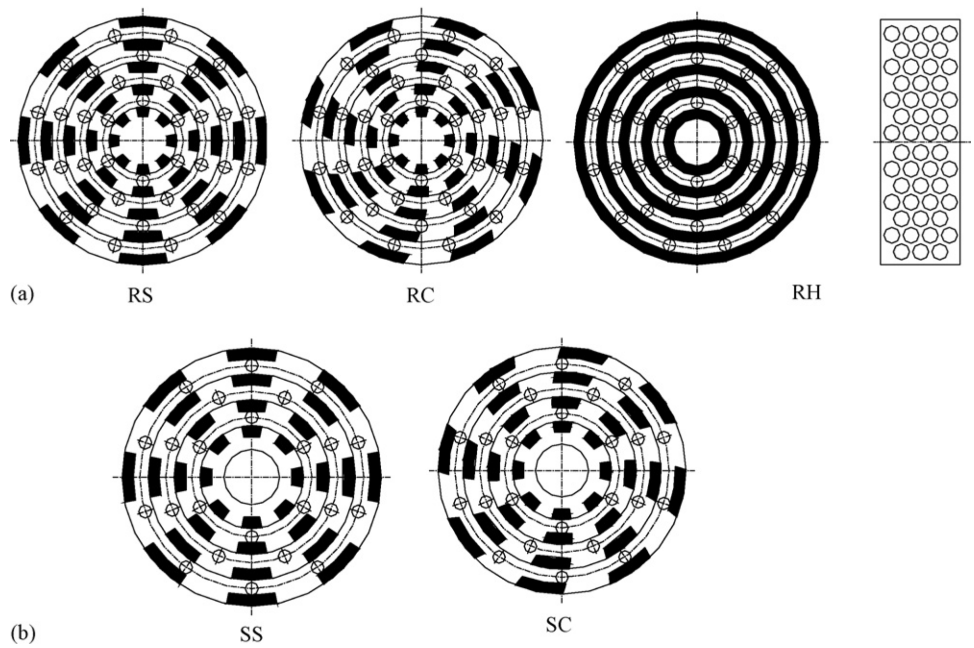

Fig. 2. Schematic description of rotor and stator. (a) Rotors used in experiments; (b) stators used in experiments.

The test procedure consists of pumping a small quantity of the said solution B (containing sulfuric acid in stoichiometric deficiency) and solution A into the RSR simultaneously after confirming the valves 5 and 8 closed (see Fig. 3). The flowrate of the solutions A and B was fixed at 300 and 40 $\mathrm{L\,h^{-1}}$, respectively. The temperature of solutions were controlled within the range of 20–25 °C by introducing circulation water into the coils, and the practical temperature of solution were measured at measuring ports ‘b’ and ‘c’. The samples were taken from sampling port ‘d’. The amount of I_3^- produced was determined by use of the spectrophotometer at 353 nm (UV-2501PC, Shimadzu corporation, Japan).

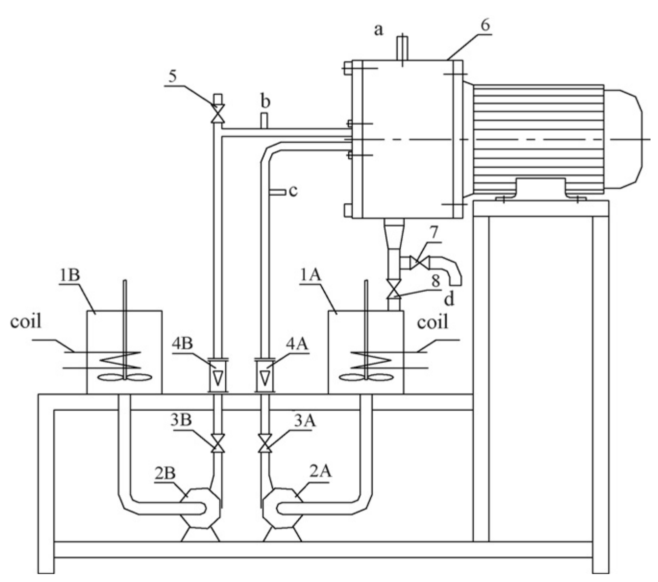

Fig. 3. Schematic diagram of experimental set-up. 1A and 1B: stirred tank; 2A and 2B: pump; 3A, 3B, 5, 7, 8: valve; 4A and 4B: rotor flowmeter; 6: rotor–stator reactor; a: gas inlet; b and c: measuring ports; d: sampling port.

3. Results and discussion

3.1. Effect of rotor speed N on X_S

Effect of rotor speed N on segregation index X_S was shown in Fig. 4 ($C_{\mathrm{H^+}} = 0.16\,\mathrm{mol\,L^{-1}}$). X_S decreases with the increase of rotor speed under experimental conditions. The reasons for this may be as follows. First, fluids were divided more fine elements with the increase of rotor speed, which makes the prob-

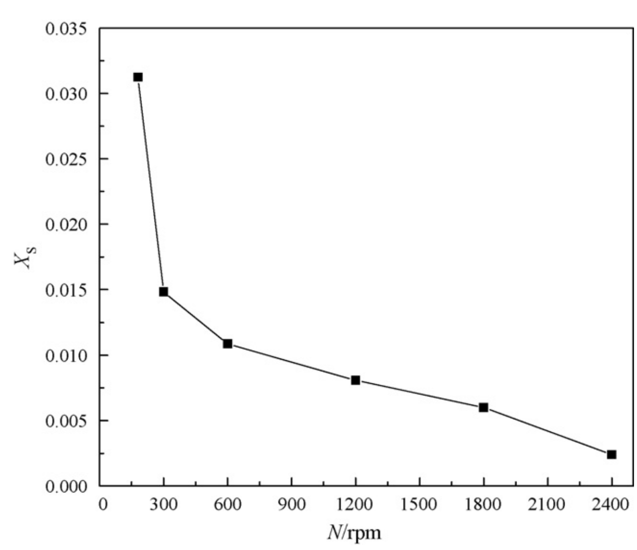

Fig. 4. Effect of rotor speed N on X_S (RS–SS combination, $C_{\mathrm{H^+}} = 0.16\,\mathrm{mol\,L^{-1}}$).

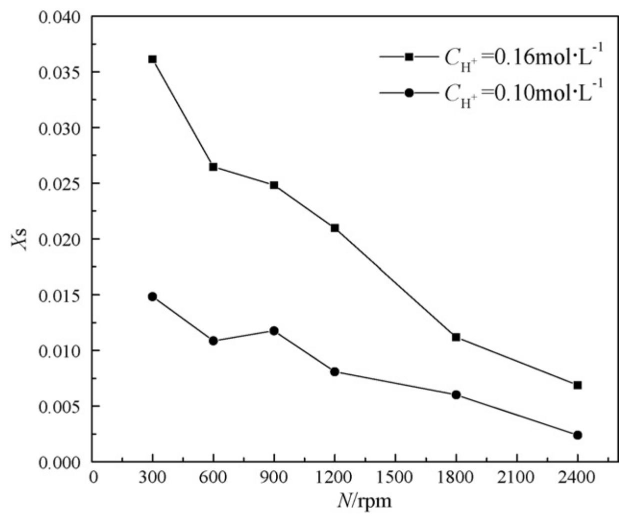

Fig. 5. Effect of reagents concentration on X_S (RS–SS combination).

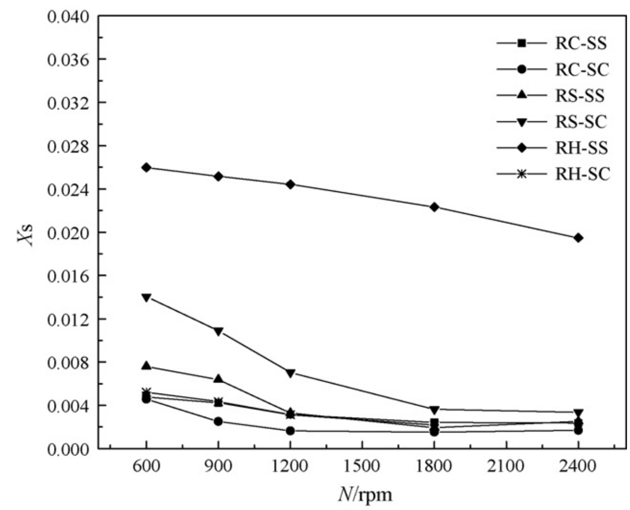

Fig. 6. Effect of rotor–stator combinations on X_S ($C_{H^+} = 0.16\,\mathrm{mol\,L^{-1}}$).

ability of local overconcentration of the injected H^+ decrease and results in good micromixing efficiency; second, relative velocity between rotor- and corresponding stator-ring became higher with increase of rotor speed, which results in fluid elements should stand higher shear force when passing through the said gaps. Therefore, the good micromixing efficiency can be obtained.

3.2. Effect of reagent concentration on X_S

When the reagents concentration were improperly selected in experiments, the amount of triiodide formed by reaction (3) may be too low or too high, which led to the optical density may not be in the range of the spectrophotometer scale. Hence, we should first select the proper reagents concentration, especially that of the acid. Fig. 5 illustrates the effects of acid concentration on X_S. A decrease of X_S was observed as the acid concentration was decreased. The acid reacts in reactions (1) and (2), the rate of the reaction (2) is more sensitive to acid concentration than that of reaction (1) because its reaction order with respect to acid is higher. When we decreased the acid concentration, the reaction (2) becomes slower notably, less iodine is formed and X_S decreased.

3.3. Effect of rotor–stator combinations on X_S

Fig. 6 reveals the influence of different rotor–stator combinations on micromixing efficiency of RSR ($C_{H^+} = 0.16\,\mathrm{mol\,L^{-1}}$). The micromixing efficiency of RH-SS is poorest and that of RC–SC is best among the investigated rotor–stator combinations. Compared with other rotor–stator combinations, fewer vortexes can be created when reagents flow through the holes opened around the circular of rotor-rings. The less vortex contribution to energy dissipation, which is favorable to mixing the reagents, leads to inferior micromixing efficiency of RH–SS combination. For stators with different structure (including SS and SC), the rotors with slots (including straight slots (RS) and curve slots (RC)) around the circular have better flexibility than the one with holes. In other words, the holed rotor is more sensitive to different structure of stator. Except the combinations of RH–SS and RS–SC, the others have similar micromixing efficiency when rotor speed was larger than 1200 rpm.

3.4. Effect of the amount of rotor- and stator-ring on X_S

The size of RSR was determined to a great extent by the amount of rotor- and stator-ring. Consequently, both the cost of RSR and the power consumption in operation were also influenced. The effect of the amount of rotor- and stator-ring on X_S was shown in Fig. 7 ($C_{H^+} = 0.16\,\mathrm{mol\,L^{-1}}$), where R*i*–S*j* means the amount of rotor- and stator-ring is *i* and *j*, respectively. When *i* was 5 and 4, and the corresponding *j* was 4 and 3, the X_S has no remarkable difference for high rotor speed (larger than 900 rpm). When *i* and *j* was less than 4 and 3, the X_S increased remarkably, i.e. micromixing efficiency is very poor. So the amount of

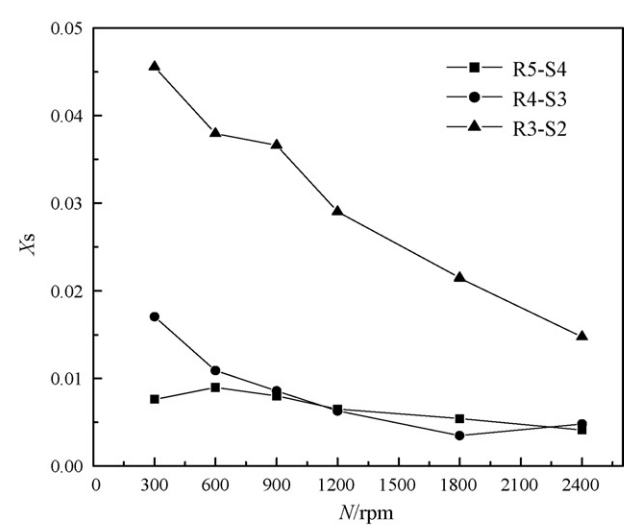

Fig. 7. Effect of the amount of rotor- and stator-ring on X_S ($C_{H^+} = 0.16\,\mathrm{mol\,L^{-1}}$).

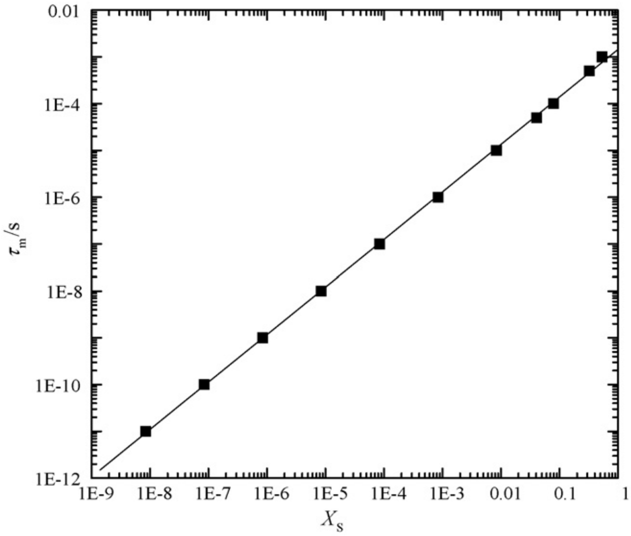

Fig. 8. Relationship between τ_m and X_S.

rings has an optimum value, which provided a basic reference for reactor design.

3.5. Determination of micromixing time τ_m

For fast chemical reaction, their characteristic reaction time τ_r is very small. The selectivity, quality or distribution of final product can be controlled only through decreasing the micromixing time τ_m less or close to τ_r. The micronmixing time τ_m cannot be calculated directly. However, it can be estimated according to experimental data. A simple and practical model named incorporation model was first developed by Villermaux et al. and used to estimate τ_m in the tank reactors [22,23]. Subsequently, this model was extended to determine τ_m of continuous reactors, e.g. couette flow reactor [8], static mixers [9] and flow cells [24]. In our previous work, the related formulas suitable for iodide–iodate reaction system were deduced and used to determine τ_m of RPB [18]. Here we also determine τ_m of RSR with this model.

Fig. 8 gives the relationship between τ_m and X_S. The logarithm correlation of them was obtained through linear regression by means of software of Microcal Origin (Version 5.0). The fit was shown as expression (6), whose correlation coefficient is 0.99983 and standard deviation is 0.05422:

$$\log \tau_m = -2.84 + 1.015 \log X_S \tag{6}$$

We substituted the data in Fig. 4 for X_S in correlation (6), a series of τ_m corresponding to the rotor speed N could be calculated. The curve of τ_m versus N was plotted in Fig. 9. The τ_m decreased as the rotor speed N increased. When the rotor speed N reached about 1800 rpm, the value of τ_m is less than 10^{-5} s, which is less than that of RPB (about 10^{-4} s).

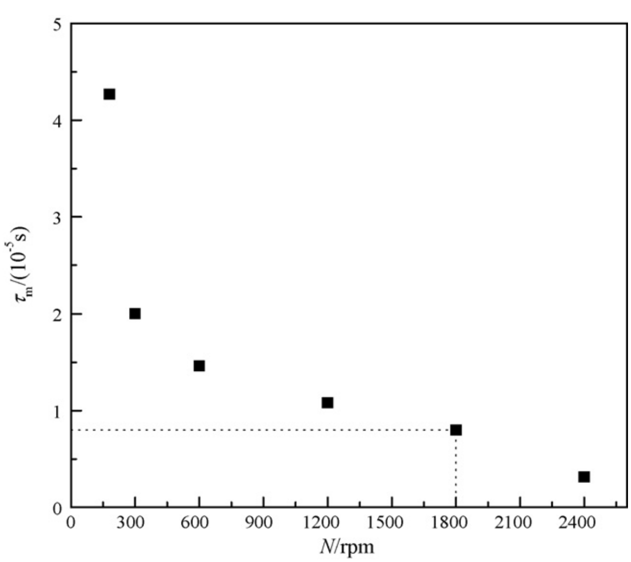

Fig. 9. Relationship between τ_m and N.

4. Conclusions

The structure and characteristics of a novel rotor–stator reactor was introduced. Employing a parallel competing iodide–iodate reaction system, the micromixing efficiency of RSR was reported. The X_S decreased with the increase of rotor speed. The decrease of injected H^+ results in the decrease of X_S. Among the six rotor–stator combinations investigated in experiment, RH-SS has poorest micromixing efficiency and that of RC–SC is best. Except the combinations of RH–SS and RS–SC, the others have similar micromixing efficiency when rotor speed was larger than 1200 rpm. At the range of conducted experiments, when the amount of rotor- and stator-ring is less than 4 and 3, respectively, the X_S increased remarkably, i.e. the micromixing efficiency becomes very poor. Based on incorporation model, the micromixing time τ_m was estimated and the relationship between τ_m and rotor speed N was reported. When the rotor speed reaches 1800 rpm, the estimated τ_m is evaluated to be less than 10^{-5} s (that of RPB is about 10^{-4} s), which proves that RSR has excellent micromixing efficiency.

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